

AD-A146 642

SYNTHESIS CHARACTERIZATION AND CRYSTAL AND MOLECULAR
STRUCTURE OF GA(CH₂S₂)₂ (U) STATE UNIV OF NEW YORK AT
BUFFALO DEPT OF CHEMISTRY R B HALLOCK ET AL. 10 OCT 84

1/1

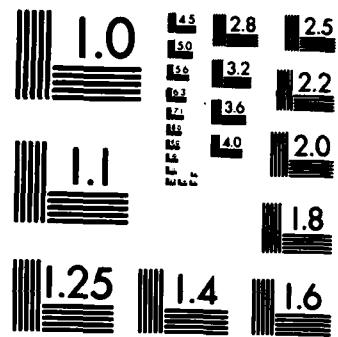
UNCLASSIFIED

TR-12 N00014-78-C-0562

F/G 7/3

NL

END



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

(P)
READ INSTRUCTIONS
BEFORE COMPLETING FORM

REPORT DOCUMENTATION PAGE		3. RECIPIENT'S CATALOG NUMBER
1. REPORT NUMBER 12	2. GOVT ACCESSION NO.	Technical Report
4. TITLE (and Subtitle) Synthesis, Characterization and Crystal and Molecular Structure of $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$		5. TYPE OF REPORT & PERIOD COVERED
7. AUTHOR(s) R. B. Hallock, W. E. Hunter, Jerry L. Atwood and O. T. Beachley, Jr.		6. PERFORMING ORG. REPORT NUMBER N-0014-78C-0562
8. PERFORMING ORGANIZATION NAME AND ADDRESS Department of Chemistry State University of New York at Buffalo Buffalo, New York 14214		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS NR-053-628
11. CONTROLLING OFFICE NAME AND ADDRESS Office of Naval Research Department of the Navy Arlington, Virginia 22117		12. REPORT DATE October 10, 1984
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		13. NUMBER OF PAGES 14
		15. SECURITY CLASS. (of this report) Unclassified
		16a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approval for Public Release, Distribution Unlimited		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) Prepared for Publication in Organometallics		
18. SUPPLEMENTARY NOTES S DTIC ELECTE OCT 16 1984 D A		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Organogallium Compounds Organogallium-Lewis base adduct X-ray structural study		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The complex $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$, prepared from $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3$, has been fully characterized by analysis, standard spectroscopic methods, cryoscopic molecular weight measurements and an X-ray structural study. The compound crystallizes in the monoclinic space group $P2_1/n$ with cell dimensions $a = 9.911(4)\text{\AA}$, $b = 16.427(6)\text{\AA}$, $c = 14.770(6)\text{\AA}$, $\beta = 90.43(4)^\circ$ and $Z = 2$ for $d_{\text{calcd}} = 1.08 \text{ g cm}^{-3}$. Full-matrix least-squares refinement led to a		

final R value of 0.075 for 1598 observed reflections. The molecule lies on a crystallographic inversion center. The geometry about the gallium atom is distorted tetrahedral with a Ga-C(av) distance of 1.99[1]Å and a Ga-N distance of 2.241(9)Å.

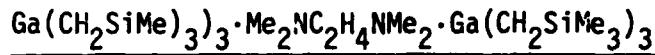


Accession For	
NTIS	GRA&I
PTIC TAB	<input checked="" type="checkbox"/>
Unannounced	<input type="checkbox"/>
Justification	
By _____	
Distribution/ _____	
Availability Codes	
Dist	Avail and/or Special
A 1	

OFFICE OF NAVAL RESEARCH
Contract N-00014-78-C-0562

Task No. NR 053-686
TECHNICAL REPORT NO. 12

Synthesis, Characterization and Crystal and Molecular Structure of



by

R. B. Hallock, W. E. Hunter, Jerry L. Atwood and
O. T. Beachley, Jr.

Prepared for Publication

in

Organometallics

State University of New York at Buffalo
Department of Chemistry
Buffalo, New York 14214

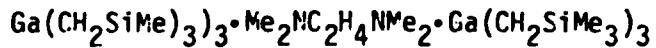
10, October 1984

Reproduction in whole or in part is permitted for any purpose of the
United States Government

*This document has been approved for public release and sale; its
distribution is unlimited

(Contribution from the Department of Chemistry, State University of New York at Buffalo; Buffalo, NY 14214 and the Department of Chemistry, University of Alabama; University, Alabama 35486.)

Synthesis, Characterization and Crystal and Molecular Structure of



by

R. B. Hallock^{1a}, W. E. Hunter^{1b}, Jerry L. Atwood^{1b} and
O. T. Beachley, Jr.*^{1a}

Abstract

The complex $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$, prepared from $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3$, has been fully characterized by analysis, standard spectroscopic methods, cryoscopic molecular weight measurements and an X-ray structural study. The compound crystallizes in the monoclinic space group $P2_1/n$ with unit cell dimensions $a = 9.911(4)\text{\AA}$, $b = 16.427(6)\text{\AA}$, $c = 14.770(6)\text{\AA}$, $\beta = 90.43(4)^\circ$ and $Z = 2$ for $d_{\text{calcd}} = 1.08 \text{ g cm}^{-3}$. Full-matrix least-squares refinement led to a final R value of 0.075 for 1598 observed reflections. The molecule lies on a crystallographic inversion center. The geometry about the gallium atom is distorted tetrahedral with a Ga-C(av) distance of $1.99[1]\text{\AA}$ and a Ga-N distance of $2.241(9)\text{\AA}$.

Introduction

The trimethylsilylmethyl group is considered to be a bulky substituent with larger steric effects than those of a methyl group. Consequently, the trimethylsilylmethyl Group 3 derivatives $M(CH_2SiMe_3)_3$ ($M=Al, Ga, In$) are considered to be weaker Lewis acids than the corresponding methyl derivatives. For example, diethylether can be readily removed from the gallium² and indium³ derivatives but the strong base NMe_3 forms stable complexes with all of these Group 3 compounds. Diamines of the type $Me_2N(CH_2)_nNMe_2$ ($n=2,3$) react with MMe_3 ($M=Al, Ga, In$) to form 1:2 complexes⁴ $Me_3M \cdot Me_2N(CH_2)_nNMe_2 \cdot MMe_3$. Cryoscopic molecular weight measurements of these methyl complexes in benzene solution are consistent with monomeric behavior and little dissociation into 1:1 complexes and free Lewis acid. During the course of some of our investigations of the chemistry of $M(CH_2SiMe_3)_3$, $Ga(CH_2SiMe_3)_3$ was reacted with a large excess of $Me_2NC_2H_4NMe_2$. The isolated product $(Me_3SiCH_2)_3Ga \cdot Me_2NCH_4NMe_2 \cdot Ga(CH_2SiMe_3)_3$ was fully characterized by analysis, standard spectroscopic methods, cryoscopic molecular weight measurements and an X-ray structural study. This structural study represents only the second report of a structure of a neutral trialkylgallium-amine donor-acceptor complex and the first with an alkyl group other than methyl.

Experimental Section

The organogallium compounds described in this investigation were oxygen and moisture sensitive and were manipulated in a vacuum line or a purified argon atmosphere. The reagent $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ was synthesized by the literature procedure.² The ligand $\text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2$ (TMED) was dried over P_2O_5 and was vacuum distilled immediately prior to reaction. Infrared spectra were recorded in the range 4000-250 cm^{-1} by means of a Perkin-Elmer 683 spectrometer. The spectra were recorded as Nujol mulls by using CsI plates. Bands due to mulling agents have been omitted. The ^1H NMR spectrum was recorded at 90 MHz and ambient temperature by using a Varian EM-390 spectrometer. Chemical shift data are reported in δ units (ppm) and are referenced to SiMe_4 as 0.00 and C_6H_6 as 7.13. Molecular weight measurements were obtained cryoscopically in benzene by using an instrument similar to that described by Shriver.⁵

Synthesis of $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$.

The title compound was prepared by vacuum distilling a large (ten fold) excess of $\text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2$ onto 0.785 g (2.37 mmol) $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ in a reaction tube equipped with a Teflon valve. After warming to room temperature, the reaction mixture was stirred for 5 h. The excess ligand was removed by vacuum distillation at room temperature to leave a viscous yellow liquid. This liquid was then heated to 45° under high vacuum and colorless crystals sublimed to the cooler portions of the tube. The crystals were isolated and resublimed at 45°. The final product $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ (0.71 g, 0.912 mmol, 77.3%) was isolated as colorless crystals: mp 89-91°C, ^1H NMR (benzene, δ) + 2.34 (s, 1.0, CH_2 -TMED), + 1.88 (s, 2.9, Me-TMED), + 0.20 (s, 11.4,

Me-CH₂SiMe₃), - 0.30 (s, 3.0, CH₂-CH₂SiMe₃); IR(Nujol, cm⁻¹) 1288 (w), 1252 (m), 1240 (s), 1162 (vw), 1128 (w), 1095 (vw), 1012 (m,sh), 999 (m), 982 (s), 960 (m), 945 (m), 860 (s,sh), 850 (s), 822 (vs), 783 (w), 748 (m), 721 (m), 677 (m), 610 (vw), 562 (w), 538 (w), 513 (m), 459 (vw); cryoscopic molecular weight; formula weight Ga(CH₂SiMe₃)₃•Me₂NC₂H₄NMe₂•Ga-(CH₂SiMe₃)₃, 778.9. Molality, obsd mol wt: 0.0990, 549; 0.0687, 491; 0.0540, 452. Hydrolysis: 0.0900 g (0.116 mmol) hydrolyzed in 6 M HCl for 48 h at 100°C gave 0.697 mmol SiMe₄, calcd 0.696. (SiMe₄ from hydrolysis was purified by fractionation through two -78°C traps and isolated at -196° and the quantity was determined by PVT measurements on the vacuum line.)

Collection of X-Ray Diffraction Data and Solution of Structure for
Ga(CH₂SiMe₃)₃•Me₂NC₂H₄NMe₂•Ga(CH₂SiMe₃)₃.

Single crystals of the air-sensitive compound were isolated by sublimation at 65°C from an attempted reaction between Ga(CH₂SiMe₃)₃ and LiH in Me₂NC₂H₄NMe₂ and were sealed under N₂ in thin-walled glass capillaries. Final lattice parameters as determined from a least-squares refinement of ((sin θ)/λ)² values for 15 reflections ($\theta > 20^\circ$) accurately centered on the diffractometer are given in Table I.

Data were collected on an Enraf-Nonius CAD-4 diffractometer by the θ-2θ scan technique. This method has been previously described.⁶ A summary of data collection parameters is given in Table I. The intensities were corrected for Lorentz and polarization effects.

Calculations were carried out with the SHELX system of computer programs.⁷ Neutral-atom scattering factors for Ga were taken from Cromer and Waber.⁸ Scattering factors stored within the SHELX program were used

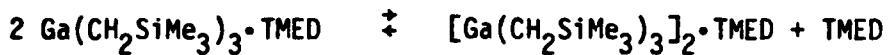
for the other atoms. The scattering factor for Ga was corrected for both the real and imaginary components of anomalous dispersion using the table of Cromer and Liberman.⁹ The position of the Ga atom was located using the MULTAN80 system of programs.¹⁰ Difference Fourier maps phased by this atom readily revealed the positions of the remaining nonhydrogen atoms. Full-matrix least-squares refinement with isotropic thermal parameters led to $R = \Sigma ||F_O| - |F_C|| / \Sigma |F_O| = 0.090$. Further refinement with anisotropic thermal parameters led to $R = 0.080$. At this point a difference Fourier map was computed and the hydrogen atoms were located. Further refinement led to final values of $R = 0.075$ and $R_w = 0.075$.

A final difference Fourier showed no feature greater than $0.6 \text{ e}/\text{\AA}^3$. The weighting scheme was based on unit weights; no systematic variation of $w(|F_O| - |F_C|)^2$ vs. $|F_O|$ or $(\sin \theta)/\lambda$ was noted. The final values of the positional parameters of atoms other than hydrogen are given in Table II. Positional parameters for all atoms (Table IV-S) and anisotropic thermal parameters (Table V-S) are deposited as Supplementary Material.

Results and Discussion

The complex $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ has been isolated from a reaction mixture containing $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ and a large excess of $\text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2$ (TMED). It is of interest that the product has both basic nitrogen atoms complexed with Lewis acids, even though the preparative reaction used an excess of TMED. Cryoscopic molecular weight measurements in benzene solution and the relatively low sublimation temperature (45-65°C) for such a large and heavy molecule suggest that the complex partially dissociates both in solution and in the vapor phase. The observed molecular weight is significantly lower than the formula weight of the compound for all solutions studied. Furthermore, the observed molecular weight is concentration dependent, decreasing with concentration. In contrast, the related complexes of the methyl metal derivatives⁴ $\text{Me}_3\text{M} \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{MMe}_3$ (M = Al, Ga, In), prepared from a reaction mixture having one mole TMED and two moles MMe₃, do not exhibit any solution properties suggestive of significant dissociation. It is also noteworthy that the melting point of $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{TMED}$ (89-91°C) is lower than that of $(\text{GaMe}_3)_2 \cdot \text{TMED}^4$ (102-103°C), which is as expected if there is partial dissociation of the former complex upon heating. The occurrence of dissociative equilibria during the isolation of the complex can also be used to account for the formation of $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{TMED}$ rather than $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{TMED}$, especially from a reaction mixture using excess TMED. If

$\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{TMED}$ is the initial product, a dissociative reaction



would lead to the formation of $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3$ and TMED. Since free TMED is the most volatile component, TMED would be removed by vacuum distillation during isolation and/or sublimation to leave $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{TMED}$, as observed.

The X-ray structural study demonstrates that the crystal consists of isolated molecules of $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{TMED}$, separated by normal van der Waals distances. There are no abnormally short intermolecular contacts. Selected interatomic distances are presented in Table III. Figure 1 shows the scheme used in labeling the atoms, while Figure 2 provides a stereoscopic view of the unit cell. The molecule lies on a crystallographic inversion center. The gallium and nitrogen atoms are in rather distorted tetrahedral environments. The gallium-alkyl distances ($\text{Ga}-\text{CH}_2\text{SiMe}_3$) are $\text{Ga}-\text{C}(1) = 2.00(1)\text{\AA}$, $\text{Ga}-\text{C}(5) = 1.99(1)\text{\AA}$ and $\text{Ga}-\text{C}(9) = 1.98(1)\text{\AA}$, the average $\text{Ga}-\text{C}(\text{sp}^3)$ distance is $1.99[1]\text{\AA}$.¹¹ These distances are shorter than the gallium-alkyl distances¹² in $\text{KGa}(\text{CH}_2\text{SiMe}_3)_3\text{H}$ which range from $2.030(9)$ to $2.003(9)\text{\AA}$, the average value being $2.029[14]\text{\AA}$. The gallium-nitrogen distance in $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{TMED}$ is $2.241(9)\text{\AA}$ which is significantly longer than that observed in the urotropine-trimethylgallium adducts,¹³ $\text{GaMe}_3 \cdot \text{C}_6\text{H}_{12}\text{N}_4$ and $(\text{GaMe}_3)_2 \cdot \text{C}_6\text{H}_{12}\text{N}_4$, $2.139(17)$ and $2.138(9)\text{\AA}$, respectively. The average $\text{Ga}-\text{C}$ bond distances in the urotropine complexes are $1.992[14]$ and $1.981[19]\text{\AA}$, respectively. For comparison, an electron diffraction study¹⁴ of

$\text{Me}_3\text{Ga}\cdot\text{NMe}_3$ reveals a Ga-N distance of $2.20(3)\text{\AA}$ and an average Ga-C distance of $1.998[4]\text{\AA}$. The angles around gallium in $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2\cdot\text{TMED}$ in decreasing order are $\text{C}(1)\text{-Ga-C}(9) = 116.9(5)^\circ$, $\text{C}(1)\text{-Ga-C}(5) = 116.6(4)^\circ$, $\text{C}(5)\text{-Ga-C}(9) = 116.2(5)^\circ$, $\text{C}(1)\text{-Ga-N}(1) = 102.1(4)^\circ$, $\text{C}(9)\text{-Ga-N}(1) = 101.0(4)^\circ$ and $\text{C}(5)\text{-Ga-N}(1) = 99.3(4)^\circ$. The Ga-C(α)-Si angles which range from $125.9(6)^\circ$ to $121.1(6)^\circ$ are substantially greater than the ideal tetrahedral angle of 109.47° . All other parameters of the molecule seem normal.

Acknowledgment. This work was supported in part by the Office of Naval Research (O.T.B.) and the National Science Foundation (Grant CHE-81-11137 (J.L.A.)).

Supplementary Material Available: Tables IV-S, V-S, VI-S, showing positional parameters, anisotropic thermal parameters observed and calculated structure factor amplitudes, (pages). Ordering information is given on any current masthead page.

References

1. (a) State University of New York at Buffalo; (b) University of Alabama
2. Beachley, O. T. Jr.; Simmons, R. G. Inorg. Chem. 1980, 19, 1021.
3. Beachley, O. T. Jr.; Rusinko, R. N. Inorg. Chem. 1979, 18, 1966.
4. Storr, A.; Thomas, B. S. Canad. J. Chem. 1970, 48, 3667.
5. Shriver, D. F. "The Manipulation of Air-Sensitive Compounds", McGraw-Hill, New York, 1969, p 159.
6. Holton, J.; Lappert, M. F.; Ballard, D. G. H.; Pearce, R.; Atwood, J. L.; Hunter, W. E. J. Chem. Soc., Dalton Trans. 1979, 45.
7. SHELX, a system of computer programs for X-ray structural studies by G. M. Sheldrick, University of Cambridge, Cambridge, England, 1976.
8. Cromer, D. T.; Waber, J. T. Acta Crystallog. 1965, 18, 104.
9. Cromer, D. T.; Liberman, D. J. Chem. Phys. 1970, 53, 1891.
10. MULTAN 80, a system of computer programs for the application of direct methods by G. Germain, P. Main, and M. M. Woolfson.
11. Esd's of average values, calculated by the "scatter formula" below, are enclosed in square brackets.

$$[\sigma] = \left[\frac{1}{N} \sum_{i=1}^{N-1} (\bar{d} - d_i)^2 \right]^{1/2}$$

12. Hallock, R. B.; Beachley, O. T. Jr.; Li, Y.-J.; Sanders, W. M.; Churchill, M. R.; Hunter, W. E.; Atwood, J. L. Inorg. Chem. 1983, 22, 3683.
13. Krause, H.; Sille, K.; Hausen, H.-D.; Weidlein, J. J. Organometal. Chem. 1982, 235, 253.
14. Golubinskaya, L. M.; Golubinskii, A. V.; Mastryukov, V. S.; Vilkov, L.V.; Bregadze, V. I. J. Organometal. Chem. 1976, 117, C4.

Table I. Crystal Data for $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2\cdot\text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2$

comp	$\text{Ga}_2\text{Si}_6\text{N}_2\text{C}_{30}\text{H}_{82}$
mol wt	778.90
space group	$\text{P}2_1/n$
cell constants	
a, Å	9.911(4)
b, Å	16.427(6)
c, Å	14.770(6)
β, deg	90.43(4)
cell vol, Å ³	2404.6
molecules/unit cell (Z)	2
ρ (calcd) g cm ⁻³	1.08
μ (calcd), cm ⁻¹	13.46
radiation	MoK α
max crystal dimens, mm	0.30 x 0.20 x 0.15
scan width, deg	0.8 + 0.2 tan θ
std reflctns	040, 004
decay of stds	< 3%
reflctns measd	2519
2θ range	2 - 40
obsd reflctns	1598
no. of parameters varied	181
GOF	2.99
R	0.075
R _w	0.075

Table II
Final Fractional Coordinates for $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2$

Atom	x/a	y/b	z/c	U(eav)
Ga(1)	0.507(1)	0.14989(9)	0.3729(8)	0.050
C(1)	0.779(1)	0.0660(7)	0.3706(8)	0.051
Si(1)	0.945	0.1380(2)	0.3639(2)	0.061
C(2)	0.975(2)	0.2084(9)	0.4600(1)	0.090
C(3)	1.085(1)	0.0621(1)	0.370(1)	0.090
C(4)	0.967(2)	0.196(1)	0.3557(1)	0.101
C(5)	0.625(1)	0.2699(8)	0.2996(8)	0.061
C(6)	0.549(5)	0.4674(3)	0.3569(3)	0.084
C(7)	0.564(2)	0.4505(8)	0.376(1)	0.106
C(8)	0.535(2)	0.332(1)	0.375(2)	0.140
C(9)	0.534(5)	0.361(1)	0.215(1)	0.259
C(10)	0.460(1)	0.1150(6)	0.2915(3)	0.067
C(11)	0.491(4)	0.0711(3)	0.2977(2)	0.088
C(12)	0.523(2)	0.0397(9)	0.1855(1)	0.095
C(13)	0.523(1)	0.1634(9)	0.1803(9)	0.084
C(14)	0.523(1)	0.079(1)	0.1846(1)	0.116
C(15)	0.457(2)	0.1167(6)	0.5085(6)	0.050
	0.0346(1)	0.0339(8)	0.5148(9)	0.080
	0.411(1)	0.1269(9)	0.5787(8)	0.083
		0.176(1)	0.5277(9)	0.091

Table III
 Bond Lengths (Å) and Bond Angles (deg) for
 $[\text{Ga}(\text{CH}_2\text{SiMe}_3)_3]_2 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2$

Atoms	Distance	Atoms	Distance
$\text{Ga}(1) \text{-- C}(1)$	2.00(1)	$\text{Ga}(1) \text{-- C}(5)$	1.99(1)
$\text{Ga}(1) \text{-- C}(9)$	1.98(1)	$\text{Ga}(1) \text{-- N}(1)$	2.241(9)
$\text{Si}(1) \text{-- C}(1)$	1.87(1)	$\text{Si}(1) \text{-- C}(2)$	1.86(1)
$\text{Si}(1) \text{-- C}(3)$	1.86(1)	$\text{Si}(1) \text{-- C}(4)$	1.85(1)
$\text{Si}(2) \text{-- C}(5)$	1.83(1)	$\text{Si}(2) \text{-- C}(6)$	1.85(1)
$\text{Si}(2) \text{-- C}(7)$	1.85(2)	$\text{Si}(2) \text{-- C}(8)$	1.81(2)
$\text{Si}(3) \text{-- C}(5)$	1.85(1)	$\text{Si}(3) \text{-- C}(10)$	1.87(2)
$\text{Si}(3) \text{-- C}(13)$	1.85(1)	$\text{Si}(3) \text{-- C}(12)$	1.86(1)
$\text{C}(13) \text{-- C}(13)$	1.47(3)	$\text{C}(13) \text{-- N}(1)$	1.51(2)
$\text{C}(14) \text{-- N}(1)$	1.52(2)	$\text{C}(15) \text{-- N}(1)$	1.50(2)

Atoms	Angle	Atoms	Angle
$\text{C}(1) \text{-- Ga}(1) \text{-- C}(5)$	116.6(4)	$\text{C}(1) \text{-- Ga}(1) \text{-- C}(9)$	116.9(5)
$\text{C}(5) \text{-- Ga}(1) \text{-- C}(9)$	116.2(5)	$\text{C}(1) \text{-- Ga}(1) \text{-- N}(1)$	102.1(4)
$\text{C}(5) \text{-- Ga}(1) \text{-- N}(1)$	99.3(4)	$\text{C}(9) \text{-- Ga}(1) \text{-- N}(1)$	101.0(4)
$\text{C}(1) \text{-- Si}(1) \text{-- C}(2)$	112.1(6)	$\text{C}(1) \text{-- Si}(1) \text{-- C}(3)$	110.2(6)
$\text{C}(2) \text{-- Si}(1) \text{-- C}(3)$	105.9(7)	$\text{C}(1) \text{-- Si}(1) \text{-- C}(4)$	112.9(6)
$\text{C}(2) \text{-- Si}(1) \text{-- C}(4)$	108.0(7)	$\text{C}(3) \text{-- Si}(1) \text{-- C}(4)$	107.3(7)
$\text{C}(5) \text{-- Si}(2) \text{-- C}(6)$	111.1(7)	$\text{C}(5) \text{-- Si}(2) \text{-- C}(7)$	113.4(8)
$\text{C}(6) \text{-- Si}(2) \text{-- C}(7)$	106.1(9)	$\text{C}(5) \text{-- Si}(2) \text{-- C}(8)$	109.2(8)
$\text{C}(6) \text{-- Si}(2) \text{-- C}(3)$	109.8(9)	$\text{C}(7) \text{-- Si}(2) \text{-- C}(8)$	105(1)
$\text{C}(9) \text{-- Si}(3) \text{-- C}(10)$	110.3(6)	$\text{C}(9) \text{-- Si}(3) \text{-- C}(11)$	112.2(6)
$\text{C}(10) \text{-- Si}(3) \text{-- C}(11)$	103.3(7)	$\text{C}(9) \text{-- Si}(3) \text{-- C}(12)$	111.2(7)
$\text{C}(10) \text{-- Si}(3) \text{-- C}(12)$	100.4(8)	$\text{C}(11) \text{-- Si}(3) \text{-- C}(12)$	108.2(7)
$\text{Ga}(1) \text{-- C}(1) \text{-- Si}(1)$	121.1(6)	$\text{Ga}(1) \text{-- C}(5) \text{-- Si}(2)$	125.9(6)
$\text{Ga}(1) \text{-- C}(9) \text{-- Si}(3)$	122.5(6)	$\text{Ga}(1) \text{-- N}(1) \text{-- C}(13)$	115.9(7)
$\text{Ga}(1) \text{-- N}(1) \text{-- C}(14)$	106.2(7)	$\text{C}(13) \text{-- N}(1) \text{-- C}(14)$	112(1)
$\text{Ga}(1) \text{-- N}(1) \text{-- C}(15)$	106.6(7)	$\text{C}(13) \text{-- N}(1) \text{-- C}(15)$	105(1)
$\text{C}(14) \text{-- N}(1) \text{-- C}(15)$	109(1)		

Figures

Figure 1 Labeling of atoms in $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$.

(Trimethylsilyl groups shown as point atoms for clarity; Ortep-II diagram showing 30% probability contours of the thermal vibration ellipsoids of non-hydrogen atoms.)

Figure 2 Stereoscopic view of unit cell of $\text{Ga}(\text{CH}_2\text{SiMe}_3)_3 \cdot \text{Me}_2\text{NC}_2\text{H}_4\text{NMe}_2 \cdot \text{Ga}(\text{CH}_2\text{SiMe}_3)_3$

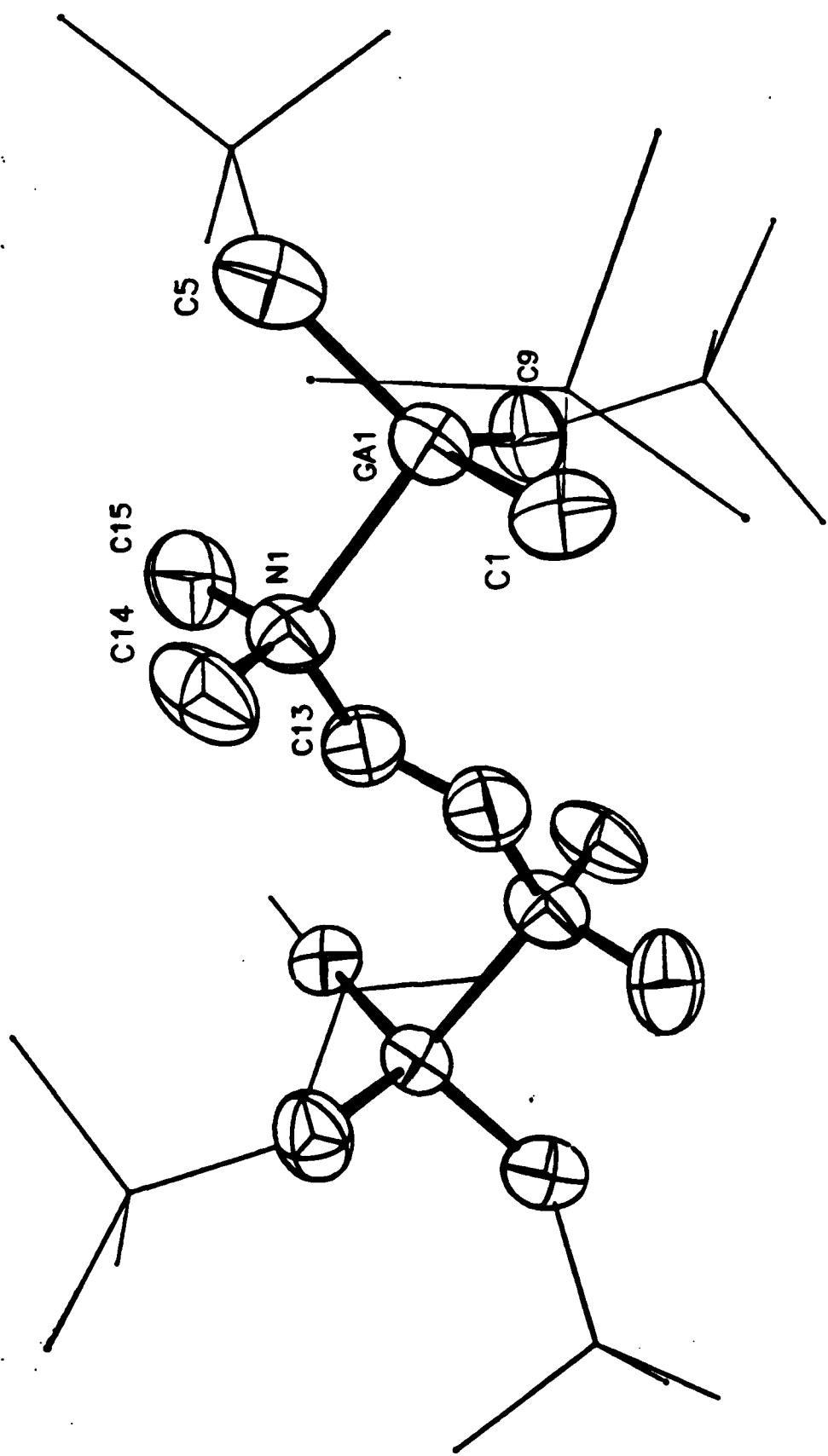


Figure 1

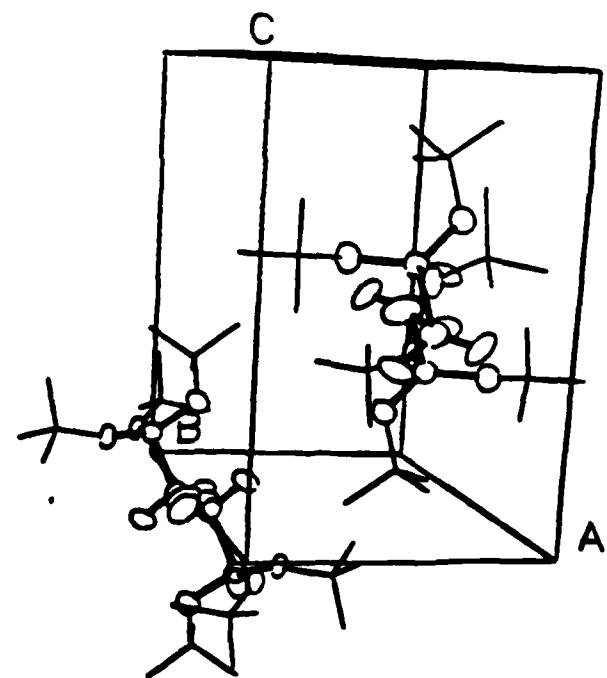
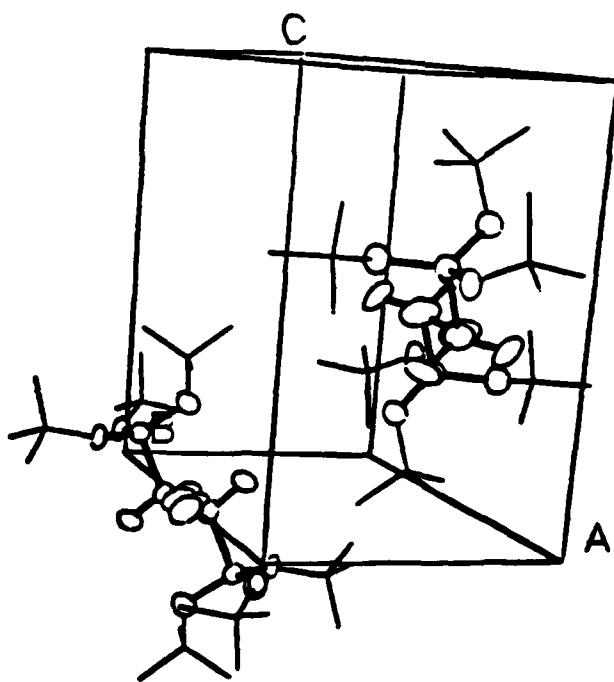


Figure 2

TECHNICAL REPORT DISTRIBUTION LIST, GEN

	<u>No.</u> <u>Copies</u>		<u>No.</u> <u>Copies</u>
Office of Naval Research Attn: Code 413 800 N. Quincy Street Arlington, Virginia 22217	2	Naval Ocean Systems Center Attn: Technical Library San Diego, California 92152	1
ONR Pasadena Detachment Attn: Dr. R. J. Marcus 1030 East Green Street Pasadena, California 91106	1	Naval Weapons Center Attn: Dr. A. B. Amster Chemistry Division China Lake, California 93555	1
Commander, Naval Air Systems Command Attn: Code 310C (H. Rosenwasser) Washington, D.C. 20360	1	Scientific Advisor Commandant of the Marine Corps Code RD-1 Washington, D.C. 20380	1
Naval Civil Engineering Laboratory Attn: Dr. R. W. Drisko Port Hueneme, California 93401	1	Dean William Tolles Naval Postgraduate School Monterey, California 93940	1
Superintendent Chemistry Division, Code 6100 Naval Research Laboratory Washington, D.C. 20375	1	U.S. Army Research Office Attn: CRD-AA-IP P.O. Box 12211 Research Triangle Park, NC 27709	1
Defense Technical Information Center Building 5, Cameron Station Alexandria, Virginia 22314	12	Mr. Vincent Schaper DTNSRDC Code 2830 Annapolis, Maryland 21402	1
DTNSRDC Attn: Dr. G. Bosmajian Applied Chemistry Division Annapolis, Maryland 21401	1	Mr. John Boyle Materials Branch Naval Ship Engineering Center Philadelphia, Pennsylvania 19112	1
Naval Ocean Systems Center Attn: Dr. S. Yamamoto Marine Sciences Division San Diego, California 91232	1	Mr. A. M. Anzalone Administrative Librarian PLASTEC/ARRADCOM Bldg 3401 Dover, New Jersey 07801	1

TECHNICAL REPORT DISTRIBUTION LIST, 053

Dr. M. F. Hawthorne
Department of Chemistry
University of California
Los Angeles, California 90024

Dr. D. Venezky
Chemistry Division
Naval Research Laboratory
Washington, D.C. 20375

~~Professor O. T. Beachley
Department of Chemistry
State University of New York
Buffalo, New York 14214~~

Dr. A. Cowley
Department of Chemistry
University of Texas
Austin, Texas 78712

Dr. W. Hatfield
Department of Chemistry
University of North Carolina
Chapel Hill, North Carolina 27514

Professor Richard Eisenberg
Department of Chemistry
University of Rochester
Rochester, New York 14627

Professor K. Neidenzu
Department of Chemistry
University of Kentucky
Lexington, Kentucky 40506

Dr. T. Marks
Department of Chemistry
Northwestern University
Evanston, Illinois 60201

Dr. J. Zuckerman
Department of Chemistry
University of Oklahoma
Norman, Oklahoma 73019

Professor K. M. Nicholas
Department of Chemistry
Boston College
Chestnut Hill, Massachusetts 02167

Professor R. Neilson
Department of Chemistry
Texas Christian University
Fort Worth, Texas 76129

Professor M. Newcomb
Department of Chemistry
Texas A&M University
College Station, Texas 77843

Professor R. Wells
Department of Chemistry
Duke University
Durham, North Carolina 27706

Professor K. O. Christe
Rockwell International
Canoga Park, California 91304

END

FILMED

11-84

DTIC